

# Assessment of the Biological Durability of Oil Palm Trunk Modified With 1,3-Dimethylol-4,5-Dihydroxyethyleneurea (DMDHEU) following Subsequent Curing at Elevated Temperatures

Siti Nurul Ashikin Rosli,<sup>a,b</sup> Kit Ling Chin,<sup>a,\*</sup> Chuan Li Lee,<sup>a,\*</sup> Mariusz Maminski,<sup>c</sup> Luqman Chuah Abdullah,<sup>a</sup> and Renata Toczyłowska-Maminska<sup>d</sup>

The biological durability of oil palm trunk modified with 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) was tested after subsequent curing at elevated temperatures. The resistance against mold, decay fungi, and subterranean termites was examined. Dimethylol dihydroxyethyleneurea-modified OPT demonstrated improved resistance against mold and decay fungi; however, the effect was lesser on subterranean termite attacks. Oil palm trunk modified with 34% DMDHEU and cured at 160 °C exhibited the most effective reduction in mass loss due to biological degradation compared to the untreated controls. High curing temperature (> 180 °C) caused cracks and ruptured the cell walls, exposing the untreated zone of the OPT to biological attack. As a non-biocidal wood modification, homogeneous dispersion of DMDHEU with adequate concentration and curing temperature is important to fully prevent OPT degradation by wood-destroying fungi and termites. These results provide valuable information for technologists to enhance or streamline the experimental design of combined modification with DMDHEU and thermal treatment, supporting their practical goals. It is recommended to continue research on the leaching of DMDHEU-modified OPT to understand the prevailing effect of DMDHEU on the biological durability of OPT, whether it is due to cross-linking or filler dispersion.

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*Contact information:* a: Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia; b: Faculty of Forestry and Environment, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia; c: Institute of Wood Sciences and Furniture, Department of Technology and Entrepreneurship in Wood Industry, Warsaw University of Life Sciences, 159 Nowoursynowska St, 02-776, Warsaw, Poland; d: Institute of Biology, Department of Physics and Biophysics, Warsaw University of Life Sciences, Nowoursynowska 166 St., bldg. 37, room P/3, 02-787 Warsaw, Poland;

\* Corresponding authors: c\_kitling@upm.edu.my; chuanli@upm.edu.my

## INTRODUCTION

Wood modification presents a biocide-free and non-toxic solution as an alternative to conventional wood preservation methods. It also enhances specific wood qualities, making it suitable for outdoor use. Various techniques for wood modification result in chemical changes within the material, achieved by employing chemical agents (chemical modification) or subjecting it to controlled degradation processes through heat application (thermal modification). The water-soluble glyoxal resin, 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU), has been widely employed as the basis for low-maintenance

cross-linking finishes in textile applications, particularly with cellulose and cellulose blended fabrics (Emmerich *et al.* 2020a). DMDHEU is a urea-based resin with additional hydroxymethyl groups that enhance cross-linking and bonding properties. Compared to urea formaldehyde (UF) resin, DMDHEU produces lower formaldehyde emissions during curing and use (Mamiński *et al.* 2018), making it a more environmentally friendly choice. Generally, DMDHEU provides better water resistance and thermal stability compared to UF, making it suitable for applications in humid environments (Mamiński *et al.* 2018). UF resin also is considered more brittle compared to DMDHEU, which typically offers better flexibility and durability in applications such as textiles (Emmerich and Militz 2020). DMDHEU is often used in textile finishing for its wrinkle-resistant properties, while UF is more common in wood adhesives and resins.

Wood modification using DMDHEU has been the subject of extensive research recently, with findings indicating improved dimensional stability and increased surface hardness without causing substantial optical alterations such as discoloration. Furthermore, DMDHEU-modified wood exhibits good resistance to weathering (Emmerich *et al.* 2020b, 2021). Treatment of wood strips using DMDHEU helps to reduce the breakdown of lignin and cellulose, thus improving the durability of the wood cell walls when exposed to simulated weathering (Xie *et al.* 2005; Brischke *et al.* 2024). Rabe *et al.* (2020) also found that treating beech with DMDHEU to crosslink cellulose microfibrils results in a stronger charring mechanism compared to untreated beech. This process can improve characteristics important for indoor items that encounter moisture (such as flooring and kitchen counters), and outdoor products subjected to diverse weather conditions. It also helps protect against moisture-related dimensional changes and deterioration caused by wood-destroying microorganisms. Thus, emphasizing DMDHEU modification could enhance the qualities of “less appropriate” wood types for usage, substituting more suitable but threatened and costly species like tropical hardwoods.

Previous research on DMDHEU modification technology predominantly concentrated on poplar, pine sapwood, and beech (Yuan *et al.* 2013; Kurt and Tomak 2019; Emmerich *et al.* 2020a; Li *et al.* 2020; Sivrikaya and Can 2022). Drying and curing methods for impregnated wood need to be adjusted, particularly when transitioning from softwood to hardwood or non-wood species. The goal is to achieve consistent curing properties and an even distribution of modification agents (Li *et al.* 2020; Goli *et al.* 2023). Modification of solid wood is typically limited to porous wood species that can be easily impregnated. In this research, DMDHEU wood modification was implemented on oil palm trunk, a plentiful waste product from the felling of oil palm trees available throughout the year. As there are approximately 40 million acres of palm plantations worldwide, exploring potential uses for oil palm trunk is crucial for sustainable development within the industry. The structure of the oil palm trunk differs significantly from that of hardwood and softwood due to its monocotyledonous nature. Oil palm trunk is considered a porous wood species capable of efficient liquid uptake. However, it contains parenchymatous tissue with starch granules in its cells, which could potentially inhibit permeability, water diffusion, and liquid transport (Wong *et al.* 2022). The latter can result in fluctuations in the chemical absorption and distribution within OPT specimens. Limited information is available regarding the impact of DMDHEU modification on OPT, particularly its biological durability.

Significant improvements in mechanical properties of OPT were observed in the authors' previous study, with a 3.8-fold increase in Modulus of Rupture (MOR) and a 3.6-fold increase in Modulus of Elasticity (MOE) for the 34% DMDHEU treatment, and a 3.2-fold increase in MOR and a 4.11-fold increase in MOE for the 17% DMDHEU treatment (Mamiński *et al.* 2016). Building on these findings, the current study aims to investigate the biological durability of OPT treated with these same concentrations. It is proposed that understanding the effects of DMDHEU at these concentrations on biological durability will provide valuable insights and further the field of wood modification research.

There is limited comprehension regarding the impact of DMDHEU treatment on the properties of OPT, as well as the permanence of its effects on biological durability and the influence of variations in treatment concentration and curing temperature on material performance. Several studies have proposed a lower curing temperature below 100 °C for DMDHEU-modified wood using various catalysts (Yuan *et al.* 2013; Li *et al.* 2020). In practice, a curing temperature of at least 160 °C is commonly used in the DMDHEU treatment of cotton fabrics (Dhiman and Chakraborty 2017; Thi *et al.* 2020). Some scholars have also proposed that subjecting OPT to heat treatment at temperatures of 140 °C or higher can notably enhance its resistance to biological degradation (Lee *et al.* 2020; Murda *et al.* 2021). In this study, higher temperatures were used to cure DMDHEU-impregnated OPT to investigate the combined impact of chemical treatment (with DMDHEU) and thermal modification (with elevated curing temperatures of 140, 160, and 180 °C) on the assessment of biological durability.

The modification process occurred in two stages: (i) wood impregnation with a solution of monomer through vacuum-pressure-impregnation, and (ii) the reagent was dried and polymerized (cured) at temperatures exceeding 140 °C. Complete impregnation of OPT is essential, and the curing stage is crucial for determining the final product quality as it affects both curing quality and the fixation of the reagent. The objectives of this research were: (i) to investigate higher curing temperatures (140, 160, or 180 °C) in conjunction with different concentrations of DMDHEU (17% and 34%) using p-toluenesulfonic acid (p-TSA) as the catalyst; (ii) to assess the impact of elevated curing temperatures on the physical properties and biological durability of OPT modified with DMDHEU; and (iii) to examine the correlation between physical properties and biological durability of the modified OPT in order to enhance understanding of how DMDHEU functions in resisting subterranean termite attacks and inhibiting the growth of mold and decay fungi on OPT. Based on these objectives, it is hypothesized that OPT modified with DMDHEU and subsequently cured at elevated temperatures will exhibit significantly increased resistance to biological degradation compared to untreated oil palm trunk. Elevated curing temperature may enhance cross-linking reactions between DMDHEU and OPT, resulting in a more stable chemical structure that is less susceptible to biological degradation. It is expected that higher concentrations of DMDHEU (34% *vs.* 17%) will result in increased cross-linking density during curing, thereby improving the biological durability of the modified oil palm trunk. Increased DMDHEU availability can lead to more extensive cross-linking reactions, contributing to better protection against biological threats. The biological durability of DMDHEU-modified OPT will be influenced by the inherent structural properties of the OPT, with cross-linking reactions enhancing these properties under specific curing temperature. The interaction between the OPT natural structure and the cross-linking provided by DMDHEU may lead to synergistic effects that bolster resistance to decay and termite attack.

## EXPERIMENTAL

### Modification of OPT with DMDHEU

The 1,3-dimethylol-4,5 dihydroxyethyleneurea (DMDHEU) was manufactured by BASF Chemicals, Germany (pH 4.3; solids 34%; viscosity 27 mPas). The research utilized an oil palm trunk (OPT) with an approximate density of  $400 \pm 20 \text{ kg m}^{-3}$ . The trunk of a 30-year-old oil palm was obtained from an oil palm plantation in Selangor, Malaysia during replanting activities. OPT specimens of size  $70 \times 20 \times 10 \text{ mm}$  and  $25 \times 25 \times 25 \text{ mm}$  were dehydrated at a temperature of  $103 \text{ }^\circ\text{C}$  until they reached an oven-dry state, then weighed. Subsequently, the specimens were kept in an environment with a temperature of  $23 \text{ }^\circ\text{C}$  and a humidity level of 65% for no less than thirty days before further processing. The modification of the oil palm trunk with DMDHEU followed the method outlined by Mamiński *et al.* (2016) in a previous study with slight modifications to the curing procedures. Vacuum impregnation processes were carried out at a pressure of 0.01 MPa for 45 min using either 17% or 34% concentration of DMDHEU with 5% p-toluenesulfonic acid (p-TSA) as the catalyst. After treatment, the excess solution was removed from the specimens using filter paper. The DMDHEU impregnated OPT samples were then placed in a conditioning room at  $23 \text{ }^\circ\text{C}$  and 65% relative humidity (RH) for 24 h prior to the curing phase to prevent potential cracks during this period. Following this, the DMDHEU impregnated samples underwent fixation at curing temperatures of 140, 160, and  $180 \text{ }^\circ\text{C}$  by placing them in an oven for 24 h, respectively. The final weights and dimensions were recorded after this step. The modified samples were stored in a conditioned room for four weeks under conditions of  $23 \text{ }^\circ\text{C}$  temperature and 65% relative humidity before being subjected to subsequent testing.

### Determination of Physical Properties of Modified OPT

Thickness swelling (TS) was measured according to the ASTM D1037-12 standard. Calculation of water absorption was performed using Eq. 1,

$$\text{WA} = (M_n - M_s) / M_s \times 100\% \quad (1)$$

where WA represents the water absorption percentage,  $M_n$  signifies the specimen's weight after immersion, and  $M_s$  denotes the specimen's weight before immersion. The immersing process was carried out using deionized water.

Weight percent gain serves as a predictor for the absorption of the altering substance. It was determined using Eq. 2,

$$\text{WPG} = (M_1 - M_0) / M_0 \times 100\% \quad (2)$$

where  $M_0$  is the initial oven-dry weight of a specimen (g), and  $M_1$  is the oven-dry weight of a specimen after treatment.

For morphological evaluation under the scanning electron microscope (SEM), the OPT specimens were softened in a water bath at  $60 \text{ }^\circ\text{C}$  for 30 min before cutting the cross-sectional surface with a sliding microtome. An EM-30AX SEM (S-3400 N, Hitachi, Japan) in the range of 10 to 20 kV acceleration voltage was used to observe the DMDHEU penetration into the cell wall. The small specimens were coated with gold-palladium and mounted on a specimen holder.

## Analysis of Fourier-Transform Infrared Spectrometry

Fourier-transform infrared (FTIR) spectra of untreated and DMDHEU-modified OPTs were obtained using a Fourier transform infrared spectroscope (FTIR- Perkin Elmer Spectrum 100-IR, Tokyo, Japan). The samples were crushed into powder form and were inserted into the FTIR chamber. The spectra were recorded at a resolution from 4000 to 700  $\text{cm}^{-1}$  in the mid-infrared region.

## Assessment of Biological Durability

*Assessment of the effectiveness of DMDHEU modification on OPT against mold in a controlled laboratory setting*

The specimens measuring  $70 \times 20 \times 10$  mm were subjected to a solution of mold (*Penicillium* spp.) spores with around  $12 \times 10^4$  spores/ mL. Inoculating the specimen with spore suspensions was conducted in a sterile environment. The bottom of each petri dish was covered with 8 to 10 layers of absorbent paper, which were moistened with water until free moisture surfaced. Two glass rods were then placed on top of the papers, upon which the OPT specimens were positioned. Subsequently, 0.55 mL of spore suspension was spread onto the surface of the specimen. Six trials were conducted for each scenario, and then the samples were incubated at 28 °C and 85% relative humidity for four weeks to encourage fungal growth. The growth of mold in terms of area coverage and intensity was evaluated based on the rating scheme outlined in Table 1 according to the ASTM D4445-10 rating scheme.

**Table 1.** The Rating Scheme Used to Evaluate the Development of Mold Growth on the Surface of the Modified OPT Specimens

| Rating | Percentage Area of Growth                   | Intensity of Visible Growth |
|--------|---|-----------------------------|
| 0      | No growth                                   | Spores remain inactive      |
| 1      | Surface covered with growth of >10%         | Early phases of development |
| 2      | Surface covered with growth of 10 to 30%    | Newly generated spores      |
| 3      | Surface covered with growth of 30 to 50%    | Moderate growth             |
| 4      | Surface covered with growth of > 50%        | Plenty of growth            |
| 5      | Surface heavily covered with a dense growth | 100% coverage               |

*Assessment of the effectiveness of DMDHEU modification on OPT against decay fungi in a controlled laboratory setting*

The samples of dimensions  $25 \times 25 \times 25$  mm<sup>3</sup> were exposed to the white rot fungi, *Pycnoporus sanguineus*, based on an adaptation of the ASTM D1413-99 test protocol. The samples were placed in a jar filled with sterilized soil medium and inoculated with white rot fungi. The fungi were cultivated on untreated OPT feeder strips for one week prior to introducing the modified OPT samples into the jar. Each treatment of the modified OPT was replicated six times. A jar containing a sample of untreated OPT inoculated with fungus served as the control. The assembled jars were then kept in darkness at 28 °C and 85% RH for an incubation period of 8 weeks. Following this, the specimens were cleaned and dried, and their mass loss due to fungal degradation was measured. A rating scheme from Table 2 was used to assess the resistance categories of modified OPT based on mass loss.

**Table 2.** Resilient Classes of Modified OPT Based on Mass Loss

| Average Mass Loss (%) | Resilient Class    |
|-----------------------|--------------------|
| 0-10                  | Highly resilient   |
| 11-24                 | Resilient          |
| 25-44                 | Moderate Resilient |
| 45 and above          | Slightly Resilient |
| Source: ASTM D1413-99 |                    |

*Assessment of the effectiveness of DMDHEU modification on OPT against subterranean termites in a controlled laboratory setting*

Specimens measuring 25 × 25 × 25 mm were subjected to subterranean termites, *Coptotermes curvignathus*, using both “forced-feeding” and “choice” tests. The laboratory termite tests lasted for four weeks and followed a modified version of the ASTM D3345-22 protocol involving exposure of the test blocks to *C. curvignathus*.

In the experiments involving “force-feeding,” termites were only provided with OPT that had been modified with DMDHEU. Each test container, filled with sand, contained either DMDHEU-modified OPT or untreated OPT (as control) specimens and was exposed to subterranean termites individually. In the “choice” tests, one sample of untreated OPT (reference) and one DMDHEU-modified OPT were placed in the same test container and simultaneously exposed to subterranean termites.

During these experiments, the receptacles were kept at a constant temperature of 28 °C and a relative humidity of 80% for four weeks. The behavior of termites in each container was observed. Following the experiment, the extent of damage on OPT specimens was assessed according to Table 3. The description of the termite attack is further elaborated with details in the study by Lee *et al.* (2022), along with the survival rate of termites as per Table 4. Prior to the visual assessment, each specimen was subjected to oven drying at 103 °C until reaching a constant mass. Subsequently, the specimens were weighed both before and after being exposed to termites in order to assess the amount of OPT ingested (mass loss).

**Table 3.** Rating Scale Used for Visual Assessment of Termite Attack was Based on an Adaptation of the ASTM D3345-22

| Rating | Description     |
|--------|-----------------|
| 4      | No attack       |
| 3      | Light attack    |
| 2      | Moderate attack |
| 1      | Heavy attack    |
| 0      | Failure         |

**Table 4.** The Classification of Termite Mortality was Based on an Adaptation of the ASTM D3345-22

| Classification | Mortality percentages |
|----------------|-----------------------|
| Slightly (S)   | 0 to 33               |
| Moderate (M)   | 34 to 66              |
| Heavy (H)      | 67 to 99              |
| Complete (C)   | 100                   |

## Statistical Analysis

Statistical analysis was performed with the SPSS statistical package for Windows, version 16.0. The impact of DMDHEU concentration and curing temperature on the physical and biological durability of modified OPT was assessed using analysis of variance. Effects were deemed not statistically significant when the p-value exceeded 0.05 at a confidence level of 95%.

## RESULTS AND DISCUSSION

### Physical Properties

Chemical alteration of a lignocellulosic material typically leads to a change in density as the modifier becomes embedded within the wood structure. The percentage increase in weight can be used to quantify the amount of impregnating agent held within the modified material. Table 5 provides pertinent WPG values, indicating that both density and WPG are influenced by the concentration of DMDHEU solution and the curing temperature.

Weight percent gains measured for OPT modified with DHDHEU are indicated to correlate retention with the degradation results. The WPG of the DMDHEU was found to be higher on OPT modified with 34 % DMDHEU than with 17% DMDHEU. Thirty-four percent and 17% DMDHEU with different curing temperatures, respectively, resulted in a 30 to 45% and 16 to 26% increase in the density from the initial 400 kg/m<sup>3</sup>. However, increasing the curing temperature to 180 °C decreased the WPG regardless of the concentration of DMDHEU. The density of DMDHEU-modified OPT with 17% and 36% concentrations was found to be between 462 to 504 kg/m<sup>3</sup> and 521 to 554 kg/m<sup>3</sup>, respectively. All modified OPT samples had higher density than untreated OPT samples.

**Table 5.** Physical Properties of OPT with Combined DMDHEU and Thermal Modification

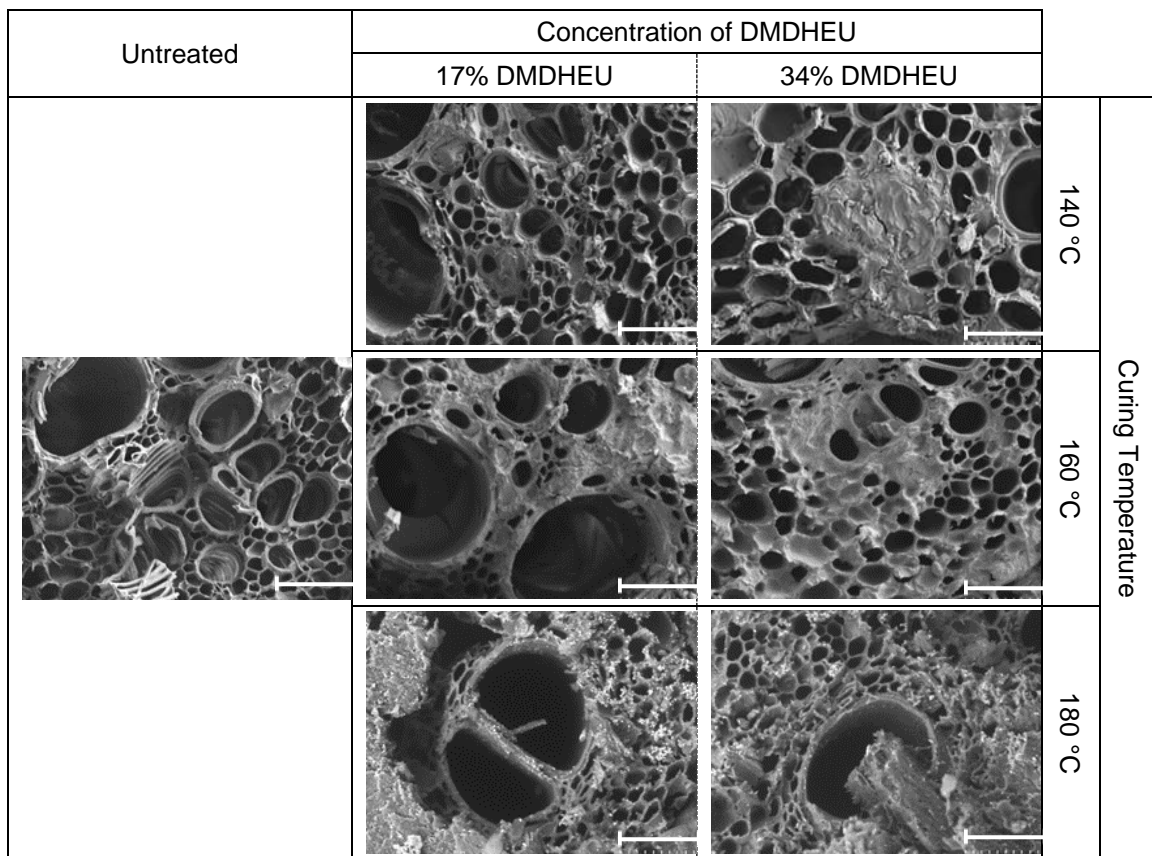
| Sample ID | Modification Treatments |                  | Weight percent gain (%) | Density (kg/m <sup>3</sup> ) | Water absorption (%) | Thickness swelling (%) |
|-----------|-------------------------|------------------|-------------------------|------------------------------|----------------------|------------------------|
|           | Concentration (%)       | Temperature (°C) |                         |                              |                      |                        |
|           | Untreated               |                  | -                       | 408 <sup>f</sup>             | 76.9 <sup>f</sup>    | 6.2 <sup>f</sup>       |
| D17T140   | 17                      | 25 <sup>f</sup>  | 25 <sup>f</sup>         | 462 <sup>e</sup>             | 56.7 <sup>e</sup>    | 3.6 <sup>e</sup>       |
| D17T160   | 17                      | 28 <sup>d</sup>  | 28 <sup>d</sup>         | 504 <sup>d</sup>             | 22.3 <sup>b</sup>    | 2.5 <sup>c</sup>       |
| D17T180   | 17                      | 26 <sup>e</sup>  | 26 <sup>e</sup>         | 498 <sup>d</sup>             | 27.6 <sup>c</sup>    | 2.7 <sup>cd</sup>      |
| D34T140   | 34                      | 37 <sup>b</sup>  | 37 <sup>b</sup>         | 554 <sup>b</sup>             | 45.1 <sup>d</sup>    | 2.8 <sup>d</sup>       |
| D34T160   | 34                      | 38 <sup>a</sup>  | 38 <sup>a</sup>         | 580 <sup>a</sup>             | 20.4 <sup>a</sup>    | 1.3 <sup>a</sup>       |
| D34T180   | 34                      | 36 <sup>c</sup>  | 36 <sup>c</sup>         | 521 <sup>c</sup>             | 28.0 <sup>c</sup>    | 2.0 <sup>b</sup>       |
|           | Pr > F                  |                  | **                      | **                           | **                   | **                     |

Note: Means followed by the same letters in the same column are not significantly different at  $p \leq 0.05$  according to Tukey's test. N.s: not significant at  $p > 0.05$ ; \*\*: significant at  $p \leq 0.05$ .

All DMDHEU-modified OPT reduced the water absorption compared to untreated OPT (Table 5). Untreated OPT showed a more rapid water uptake than modified OPT after 24 hours of submersion time. The improvement of hydrophobicity of OPT modified with DMDHEU may be due to the polymerization of DMDHEU monomers in the OPT cell walls. Oil palm trunk modified with 34% DMDHEU and further cured at 160 °C (D34T160) had the lowest water absorption (20.4%) and thickness swelling (1.3%) among

the other DMDHEU-modified OPT samples. However, further increasing the curing temperature to 180 °C did not enhance the dimensional stability of the DMDHEU-modified samples. The reduced water absorption of the wood treated with DMDHEU is attributed to the infiltration of DMDHEU into the cell walls, causing an increase in cell wall volume. This decrease in available sites within the cell wall limits its ability to absorb water molecules (Emmerich *et al.* 2020a). Additionally, altering DMDHEU reduces the point at which fibers become fully saturated by filling tiny pores that can absorb water in the untreated wood cell walls rather than reacting with the hydroxyl groups (Li *et al.* 2020).

Scanning electron microscopy images on the cross-sections of untreated and DMDHEU-modified OPT with different DMDHEU concentrations and curing temperatures are shown in Fig. 1. Untreated OPT samples exhibited a highly void structure. The empty cell wall, the pit, and the parenchyma are observed in the micrographs. The untreated OPT was covered with an uneven layer and had several void spaces. After the DMDHEU modification, the cell walls and vessels of the modified samples were filled with chemicals dispersed in the cell wall.



**Fig. 1.** Scanning electron microscopy images on the cross-sections of untreated and DMDHEU/thermal-modified OPT with different DMDHEU concentration and curing temperature. Scale bar = 100  $\mu$ m

Compared to the untreated samples, DMDHEU-modified OPT showed cells with filled lumen and cell walls, resulting in smoother lining and raised edges of pits. DMDHEU also filled some pits in the modified samples, potentially reducing water entry into the OPT and decreasing its dimensional change. The modification resulted in smaller pores in the OPT due to partial filling with DMDHEU, providing additional physical support for the



cell wall. Additionally, the hydrophobic nature of DMDHEU increased the bulkiness of the cell wall and hindered its ability to expand when absorbing water. This implies that unmodified OPT absorbs more water than modified OPT due to less space for water adsorption caused by DMDHEU filling larger pores. Higher density wood species are more challenging to absorb resin. While the tendency is not entirely exact, it may act as a guide to ascertain the suitability of a wood species for resin impregnation. Li *et al.* (2019a), reported that wood samples from *Pinus massoniana* Lamb. and *Pinus sylvestris* var. *mongolica* Litv., treated with 50% DMDHEU concentration, had minor cracks in the cell walls, with resins dispersed within the lumens. Yuan *et al.* (2013) indicated in their study that no substances were detectable in the macro-pores of *Populus ussuriensis* modified with 30% DMDHEU at a curing temperature of 90 °C; however, nitrogen compounds, identified as cured DMDHEU, were present on the cell wall at a curing temperature of 120 °C. Li *et al.* (2019b) discovered that the bamboo cell wall modified with 50% DMDHEU concentration had a smoother cross-section than untreated bamboo, despite the presence of only a few DMDHEU resin-filled cell holes. It is essential to thoroughly expose the transverse cross-section of a specimen to the solutions.

The inner wall of the D34T160 samples appeared smoother and fuller compared to the other treatment combinations, which indicated that the 34% DMDHEU penetrated the cell walls, retained, and properly cured at 160 °C. The dimensional stability significantly improved through modification of the OPT in the cell walls rather than in cell cavities. Excessive resin will function solely as a physical filler for the macropores, whilst insufficient resin may indicate a deficiency. The amount of DMDHEU found within the ring spaces of the cellular structure increased as the concentration of DMDHEU increased from 17% to 34%, showing that a higher amount of DMDHEU was retained in the modified OPT. The SEM studies indicate that raising the curing temperature from 140 to 160 °C improved the dispersion and retention of DMDHEU within the cells. Furthermore, the chemical particles of DMDHEU exhibited enhanced interfacial adhesion between the wood and the polymer. A well-dispersed modifier will result in a notable enhancement of the characteristics of the treated wood (Emmerich *et al.* 2020b).

Another significant discovery was that the cellulose wall of DMDHEU-modified OPT developed cracks at a curing temperature of 180 °C, and numerous particles of resin were dispersed within the lumens. Additionally, at this temperature, the intercellular layer demonstrated a bulk appearance with a brittle surface that had fractured. The parenchyma cells shrank, and the cellular volume decreased. In contrast, cross-sectional images of the untreated OPT sample revealed intact vascular bundles densely surrounded by intact parenchyma cells. However, in samples cured at 180 °C, parenchyma cells nearly separated from vascular bundles due to potential degradation of cell wall components. This could elucidate why WPG decreased for modified samples as temperatures reached 180 °C; an observation consistent across different concentrations of DMDHEU cured under similar conditions. Moreover, when conventional catalysts (acid or base) are used for modification with high curing temperatures, they have the potential to cause oxidative degradation or hydrolysis (Behr *et al.* 2018). It is suggested that proton concentration and curing temperature rise correspondingly with accelerate catalytic hydrolysis which may further contribute to polysaccharide cell wall component degradation (Yuan *et al.* 2013). At 140 °C, DMDHEU typically undergoes slower crosslinking, resulting in moderate adhesion and flexibility. In contrast, at 160 °C, the reaction accelerates, leading to improved interfacial bonding and potentially greater strength and durability. However, higher temperatures at 180 °C thermal degradation was observed on the modified samples.

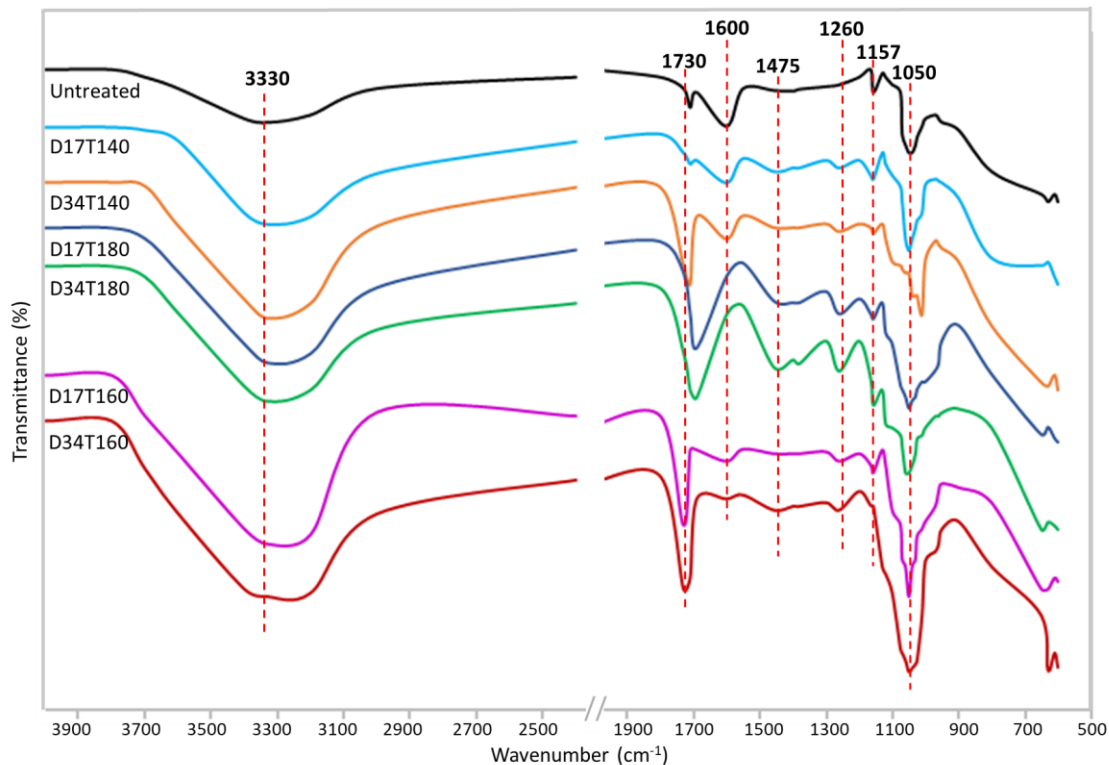
## FTIR Analysis

The FTIR analysis shown in Fig. 2 indicates changes in the modified wood samples compared to the untreated control. Specifically, the FTIR spectra of the DMDHEU-modified OPT showed a broad peak around  $3300\text{ cm}^{-1}$ , which indicates an increase in O-H groups. This rise can be attributed to the introduction of additional O-H functionalities during the reaction between DMDHEU and OPT. Although the polycondensation reaction between DMDHEU and the existing O-H groups within the OPT was ongoing, new O-H groups were likely introduced through hydrogen bonding. The broadening of the peak suggests that the modification process enhanced hydrogen bonding interactions, which played a key role in altering the chemical environment and strengthening the material's internal structure. Previous studies have also reported the O-H stretching vibration in this spectral region (González-Peña and Hale 2011; Chen *et al.* 2017; Sivrikaya and Can 2022).

Infrared spectroscopy analysis of untreated and DMDHEU-modified OPT revealed an increase in carbonyl content ( $1707$  to  $1733\text{ cm}^{-1}$ ) with higher DMDHEU concentrations. This is attributable to the carbonyl groups present in the DMDHEU compound. The peaks at  $1730\text{ cm}^{-1}$  corresponded to acetyl, carboxyl, and other carbonyl functionalities in wood hemicellulose, lignin, and related compounds (Cai *et al.* 2019; Hoffmann *et al.* 2022). The introduction of DMDHEU shifted these peaks, and the formation of an amide acetyl group ( $\text{O}=\text{C}-\text{N}$ ) was observed in the DMDHEU-modified OPT samples. A pronounced carbonyl band at  $1730\text{ cm}^{-1}$  was detected in the DMDHEU-modified OPT, overlaying the native carbonyl groups in the untreated OPT ( $1710\text{ cm}^{-1}$ ). This indicates that the carbonyl bands of DMDHEU were superimposed on those inherent to the wood. The position of the carbonyl peak maximum in DMDHEU is influenced by the electron density of the nitrogen atoms. Degradation of the reacted DMDHEU can lead to the formation of DMDHEU monomers or oligomers with terminal N-methylol groups, or further cleavage of the N-methylol group resulting in secondary amino groups like those in dihydroxyethylene urea (Xie *et al.* 2005). Unreacted, monomeric DMDHEU is known to display a peak maximum at  $1690\text{ cm}^{-1}$ , while the formation of ether bonds through condensation of the N-methylol groups causes a shift to higher wavenumbers ( $1730\text{ cm}^{-1}$ ). Dihydroxyethylene urea, a DMDHEU precursor lacking N-methylol groups, shows a peak maximum around  $1690\text{ cm}^{-1}$ , and the peak maximum for urea is assigned to  $1680\text{ cm}^{-1}$  (Verma *et al.* 2009). The shift to lower wavenumbers ( $1690\text{ cm}^{-1}$ ) can be attributed to the degradation of the ether groups in condensed DMDHEU at the high curing temperature of  $180\text{ }^{\circ}\text{C}$ , which would reduce the wavenumber of the carbonyl peak. Furthermore, the high temperatures can modify the lignin and hemicellulose in OPT, which are essential for maintaining its structural integrity. Excessive heating may consequently weaken these crucial components, rendering the OPT more susceptible to cracking.

Lignin-related bands were observed at  $1600\text{ cm}^{-1}$ ,  $1505\text{ cm}^{-1}$ , and  $1450\text{ cm}^{-1}$  ( $\text{CH}_2$ -deformation) (Ghosh *et al.* 2009). In DMDHEU-modified OPT, the aromatic stretching vibration ( $1504\text{ cm}^{-1}$ ) might have been overlaid by the  $\text{CH}_2$ -deformation vibration ( $1475\text{ cm}^{-1}$ ). Furthermore, the peak at  $1600\text{ cm}^{-1}$  was diminished in DMDHEU-modified OPT compared to the untreated sample. This suggests that chemical cross-linking may have occurred between DMDHEU and the lignin component of the OPT. The peak located at  $1475\text{ cm}^{-1}$  was attributed to  $\text{CH}_2$  deformation in the N-methylol group of DMDHEU. Meanwhile, an enhanced stretching vibration at approximately  $1260\text{ cm}^{-1}$  was detected at the DMDHEU-modified OPT, indicating the presence of DMDHEU modification. Chen *et al.* (2024), identified the stretching peak associated with the C-O bond of the carboxylate group in the range of  $1260$  to  $1240\text{ cm}^{-1}$ . This suggested that the chemicals not only filled

the cell spaces but also cross-linked with certain groups within the OPT, resulting in the formation of carboxyl groups. Furthermore, the peak of C-O-C ( $1157\text{ cm}^{-1}$ ) gradually increased due to the crosslinking reaction between OPT and DMDHEU and the condensation reaction of DMDHEU to form an ether bond (Cai *et al.* 2019).



**Fig. 2.** FTIR spectra of untreated and DMDHEU-modified OPT

The FTIR analysis revealed that the DMDHEU modification of oil palm trunk led to an increase in the band between  $1030$  and  $1060\text{ cm}^{-1}$ . This band is associated with the stretching vibrations of alcohol and ether groups in the polysaccharides. In native wood, this region is primarily attributed to the C-O stretching in the alcohol and ether groups of polysaccharides, along with a minor contribution from lignin. In the modified wood, the alcohol and ether groups of the condensed DMDHEU also contribute to this band (Yuan *et al.* 2013). The enhanced stretching vibrations in the DMDHEU-modified OPT suggest chemical crosslinking between cellulose and DMDHEU. As the curing temperature increased to  $160\text{ }^{\circ}\text{C}$ , a downward deflection at approximately  $1050\text{ cm}^{-1}$  was observed, indicating that the higher temperature facilitated the formation of alcohol and ether groups due to the reaction between cellulose and DMDHEU. The most pronounced peaks were detected for the D17T160 and D34T160 samples, suggesting a greater fixation rate of DMDHEU within the oil palm trunk (OPT) and an increased amount of alcohol and ether groups. In contrast, the peaks for the D17T180 and D34T180 samples were less intense compared to those for the D17T160 and D34T160 samples, as the higher temperature of  $180\text{ }^{\circ}\text{C}$  likely resulted in increased volatilization of the chemicals, which hindered their fixation within the OPT. Goli *et al.* (2023) found that the high thermal modification temperature ( $200$  to  $215\text{ }^{\circ}\text{C}$ ) prior to the DMDHEU impregnation process on *Fagus sylvatica* L wood caused some degradation of the hemicelluloses. This caused there to be less interaction between the cell walls with the chemical. Their study also found that

increasing the thermal modification temperatures from 120 °C to 200 °C and 215 °C resulted in less permanent swelling of the cell wall due to less embedded chemicals.

### Protection Against Mold

The DMDHEU-altered samples were exposed to a spore suspension of *Penicillium* sp. molds in controlled conditions of 95% RH and a temperature set at 28 °C. The assessment involved the examination of fungal growth and the intensity of their development, as detailed in Table 1. Each treatment condition was replicated six times. The findings from the evaluation of DMDHEU-modified OPT are outlined in Table 6.

Values measured on DMDHEU-modified OPT ranged from 1.75 (10 to 30% of growth) to 3.75 (more than 50% of growth) for the area coverage of mold and from 2 (slight) to 4.5 (strongly marked) for the intensity of mold growth. The findings indicate that applying DMDHEU to OPT inhibited mold growth and reduced its severity compared to untreated samples, which exhibited extensive mold coverage with a rating value of 5 for both area and intensity of fungal growth.

The untreated OPT demonstrated minimal resilience to mold exposure and was quickly overrun with mold after three days. In contrast, the DMDHEU-modified OPT exhibited considerable resistance to mold growth after four weeks of exposure. At the end of one month, the untreated OPT samples were completely engulfed by mold.

The surface of modified samples with 17% DMDHEU concentration at 140 °C (D17T140) exhibited a significant volume of hypha contamination. However, increasing the DMDHEU concentration to 34% and curing temperature to 160 °C, enhanced mold resistance, resulting in lower ratings for both area and intensity of mold growth after four weeks of testing (1.75 and 2, respectively). The improvements in mold resistance can be ascribed to various factors: (i) blocking the passage for mold from entering the wood through dispersion filling of DMDHEU; (ii) decreasing moisture adsorption due to DMDHEU modification inhibiting fungi growth on the surface; and (iii) altering the pH value of the wood surface by introducing an acidic catalyst during modification, rendering it unsuitable for mold growth (Nath *et al.* 2022).

**Table 6.** Ratings for Mold Growth on DMDHEU Modified OPT Specimens

| Sample ID | Modification treatments |                  | Number of samples the mould starts to grow |       |       |       |       | Rating on percentage area of growth (Day 28) | Rating on intensity of visible growth (Day 28) |
|-----------|-------------------------|------------------|--|-------|-------|-------|-------|--|--|
|           | Concentration (%)       | Curing Temp (°C) | Day 1                                      | Day 2 | Day 3 | Day 4 | Day 5 |  |  |
|           | Untreated               |                  | -  | 5     | -     | -     | -     | 5  | 5  |
| D17T140   | 17                      | 140              | -  | 2     | 3     | -     | -     | 3.75   | 4.5  |
| D17T160   | 17                      | 160              | -  | -     | 2     | 3     | -     | 3.5  | 3  |
| D17T180   | 17                      | 180              | -  | -     | 1     | 4     | -     | 3.75   | 3.25   |
| D34T140   | 34                      | 140              | -  | -     | 1     | 4     | -     | 3  | 4  |
| D34T160   | 34                      | 160              | -  | -     | -     | -     | 5     | 1.75   | 2  |
| D34T180   | 34                      | 180              | -  | -     | -     | 4     | 1     | 2.25   | 3  |

In contrast, further increasing the temperature to 180 °C caused a reduction of mold resistance on the surface of the OPT-modified samples, regardless of the DMDHEU concentrations. Although DMDHEU was penetrated to cells of modified OPT, the high curing temperature of 180 °C caused cracks and ruptured the cell walls, which caused an increase in water absorption compared to samples cured at 160 °C (Fig. 1).

## Resistance Against Decay Fungi

Resistance to decay fungi, *P. sanguineus*, was assessed after twelve weeks of exposure by measuring the mass loss of the DMDHEU-modified OPT specimens. For every treatment, six replicates were examined, and the findings are detailed in Table 7. Wong *et al.* (2022) reported that OPT contained extractable starch at levels as high as 14%, which causes it to be susceptible to fungal attack. The findings indicate that the DMDHEU alteration enhanced OPT's resistance to decay. Depending on DMDHEU concentration, the resilience of modified OPT with DMDHEU against *P. sanguineus* was between 5.7 to 20.1% for 17% concentration of DMDHEU and 18.7 to 33.6% for 34% concentration of DMDHEU in comparison with controls. Treatment with a lower DMDHEU concentration of 17% and temperature of 140 °C, did not keep OPT from fungal degradation as high mass losses were documented (40.1%), which is similar to what was observed in untreated OPT (45.8%). Higher fungal resistance was observed on modified samples modified with a higher concentration of 34% and higher temperatures of 160 and 180 °C.

**Table 7.** Mass Losses Documented on DMDHEU-Modified OPT and Control Samples after Being Exposed to *P. sanguineus* for a Period of 12 Weeks

| Sample ID   | Modification Treatments |                  | Mean Mass Loss (%) | Resilient Class    |
|---|-------------------------|------------------|--------------------|--------------------|
|   | Concentration (%)       | Curing Temp (°C) |                    |                    |
| Untreated   |                         |                  | 45.8 <sup>g</sup>  | Slightly resilient |
| D17T140   | 17                      | 140              | 40.1 <sup>f</sup>  | Moderate resilient |
| D17T160   | 17                      | 160              | 25.7 <sup>c</sup>  | Moderate resilient |
| D17T180   | 17                      | 180              | 30.1 <sup>e</sup>  | Moderate resilient |
| D34T140   | 34                      | 140              | 27.1 <sup>d</sup>  | Moderate resilient |
| D34T160   | 34                      | 160              | 12.2 <sup>a</sup>  | Resilient          |
| D34T180   | 34                      | 180              | 20.3 <sup>b</sup>  | Resilient          |
| Pr > F  |                         |                  | **                 | -                  |
| Note: Means followed by the same letters in the same column are not significantly different at $p \leq 0.05$ according to Tukey's test. n.s: not significant at $p > 0.05$ ; **: significant at $p \leq 0.05$ . |                         |                  |                    |                    |

Cell wall polymers can expand, pores can be blocked, and the size of micro-voids can be reduced through DMDHEU modification. This process reduces the amount of bound water and partially fills the pores, thereby preventing the penetration of small diffusible agents needed for fungal degradation (Kurt and Tomak 2019; Li *et al.* 2020). Wood moisture levels, the physical obstruction of fungal hyphae penetration pathways, and pH value could also impact the colonization of fungi (Pfeffer *et al.* 2011). It was shown by Ringman *et al.* (2014) that decay resistance of modified woods is due to inhibition of cell wall penetration of fungal molecules needed for oxidative degradation of wood polymers and insufficient cell wall moisture content to allow for diffusion.

Dimethylol dihydroxyethyleneurea alteration led to decreased mass losses in comparison to the control group, but it was not effective in completely protecting the entire sample from decay fungi. Throughout the decay test, fungal mycelium was present on the surface of all samples. Kurt and Tomak (2019) found that DMDHEU did not display any toxic effects on fungal development in the specimens. They also noted that the effectiveness of DMDHEU against decaying organisms operates through a different mechanism compared to traditional preservatives. The presence of voids in OPT, caused by a lower concentration of 17% DMDHEU and cracks from curing at elevated temperature (180 °C), may serve as entry points for fungi mycelium, resulting in higher moisture absorption during levels of decay testing and higher mass loss. Based on their hypothesis, an increased amount of micro-voids or cracks present on the OPT surfaces may promote fungal invasion. This can allow hyphae of wood-damaging fungi to penetrate protected surfaces and subsequently degrade untreated areas of the wooden substrate (Emmerich and Militz 2020).

### Resistance Against Subterranean Termites *Coptotermes curvignathus*

The termite survival rating (categorized as per Table 4), the wood sample degradation rating (ranging from 0 to 4 based on the rating in Table 3), and the mean of consumed wood assessed during both “forced-feeding” and “choice feeding” experiments are displayed in Table 8.

**Table 8.** Mean of Termite Survival Rates, Mean Mass of OPT Consumed, and Degradation Ratings Recorded After Four Weeks on DMDHEU-Modified OPT

| Modification Treatments |                  | Termite Mortality |                 |                    |                 | Mean Mass Loss (%) |                   | Rating on Visual Assessment of Termite Attack (Number of samples per rating) |   |   |   |   |    |   |   |   |   |
|-------------------------|------------------|-------------------|-----------------|--------------------|-----------------|--------------------|-------------------|--|---|---|---|---|----|---|---|---|---|
|                         |                  | FF                |                 | CF                 |                 | FF                 | CF                | FF   |   |   |   |   | CF |   |   |   |   |
| Concentration (%)       | Curing Temp (°C) | %                 | Classification* | %                  | Classification* |                    |                   | 4  | 3 | 2 | 1 | 0 | 4  | 3 | 2 | 1 | 0 |
|                         |                  | Untreated         |                 | 28.9 <sup>f</sup>  | S               | -                  | -                 |  |   |   |   |   |    |   |   |   |   |
| 17                      | 140              | 42.7 <sup>e</sup> | M               | 29.8 <sup>cd</sup> | S               | 38.9 <sup>f</sup>  | 28.4 <sup>f</sup> |  |   | 6 |   |   |    | 3 | 3 |   |   |
| 17                      | 160              | 45.1 <sup>e</sup> | M               | 32.3 <sup>a</sup>  | S               | 26.1 <sup>e</sup>  | 16.2 <sup>e</sup> |  |   | 4 | 2 |   |    | 2 | 4 |   |   |
| 17                      | 180              | 65.7 <sup>d</sup> | M               | 30.1 <sup>bc</sup> | S               | 20.7 <sup>c</sup>  | 12.7 <sup>d</sup> |  |   | 6 |   |   |    | 4 | 1 |   |   |
| 34                      | 140              | 70.2 <sup>c</sup> | H               | 30.4 <sup>b</sup>  | S               | 22.6 <sup>d</sup>  | 10.8 <sup>c</sup> |  | 1 | 5 |   |   |    | 6 |   |   |   |
| 34                      | 160              | 90.5 <sup>a</sup> | H               | 30.4 <sup>b</sup>  | S               | 8.2 <sup>a</sup>   | 2.9 <sup>a</sup>  | 1  | 5 |   |   |   |    | 4 | 2 |   |   |
| 34                      | 180              | 84.6 <sup>b</sup> | H               | 29.5 <sup>d</sup>  | S               | 12.8 <sup>b</sup>  | 6.5 <sup>b</sup>  |  | 4 | 2 |   |   |    | 5 | 1 |   |   |
| Pr > F                  |                  | **                |                 | **                 |                 | **                 | **                |  |   |   |   |   |    |   |   |   |   |

Note: FF = forced-feeding; CF = Choice feeding; “nr” = not relevant. Classification\* refer to Table 4. Means followed by the same letters in the same column are not significantly different at p ≤ 0.05 according to Tukey’s test. n.s: not significant at p > 0.05; \*\*: significant at p ≤ 0.05.

### Termite Mortality

Termites in containers with DMDHEU-modified OPT were initially active, creating intricate tunnels within the glass jars during the first week of the experiment.

However, their activity decreased in the second to third week, except those in the 17% concentration modified OPT. Most termites in containers with modified OPT blocks did not survive beyond the third week. In contrast, termites in control samples remained consistently active and built more complex tunnel systems throughout the experiment.

Termite mortality was heavy with the D34T160 samples (survival % = 90.5) in the forced-feeding tests, suggesting that the modified OPT contained a large proportion of undigested material when utilized as the primary food source for termites. Oil palm trunk impregnated with a lower concentration (17%) of DMDHEU had a higher survival of termites than the OPT impregnated with 34% DMDHEU. Survival rates were significantly lower in the treated OPT compared to the untreated OPT. When termites consumed both DMDHEU- modified and untreated OPT in a choice feeding test, their survival was similar to that of the control group. This suggests that ingesting DMDHEU-impregnated OPT is not directly lethal for termites as long as other food sources are present.

### **Termite Degradation of DMDHEU-Modified OPT**

The table presents the termites' survival percentage, the rating of degradation of OPT samples (ranging from 0 = strong attack to 4 = no attack as per Table 5), and the average percentage of OPT ingested during the four-week incubation termite test. Untreated OPT were rated 0 (equivalent to controls). When termites were provided with only DMDHEU- modified OPT as their food source, they exhibited a range of degradation levels in the OPT samples, from mild attack (rating 4) to severe attack (rating 1), based on the specific modification methods used. Dimethylol dihydroxyethyleneurea impregnated OPT cured at 160 °C were less degraded than OPT cured at 140 °C, which may suggest a higher curing degree at 160 °C. However, increasing the curing temperature did not prevent the degradation of termites, as all modified samples were slightly more degraded when the temperature increased to 180 °C compared to 160 °C. Samples treated with 34% DMDHEU at 160 °C (referred to as D34T160) exhibited lower degradation, indicating less susceptibility to damage (average rating of 3, minor damage). In comparison, OPT samples cured at the same temperature with a retention rate of only 17% demonstrated moderate degradation (primarily receiving ratings of 2). This observation implies a potential dose-response relationship.

Termites showed a preference for consuming untreated OPT, as indicated by the degradation rates observed when termites were presented with a choice between two food sources. Termites consumed less modified OPT when given a choice compared to being force-fed for the majority of parameters examined. The consumption rate was only equivalent for the OPT modified with 17% DMDHEU at 140 °C (D17T140), and this can be linked to a decreased level of damage observed.

When faced with a selection of food sources, subterranean termites typically showed a preference for alternatives other than DMDHEU-modified OPT, which more closely emulates their real-life feeding conditions. Among the modified samples, D34T160 can be considered a good choice among other DMDHEU modified samples protecting OPT from termite attack. Some of the OPT treated with DMDHEU showed significant deterioration but did not lead to complete termite mortality in the "forced-feeding" test, indicating low or negligible toxicity. In contrast, the "choice" tests indicated minimal OPT degradation and low termite mortality, suggesting that termites showed only a slight preference for modified OPT. As a result, indirect protection of the treated samples was achieved.

The WPG showed a main influence on the properties of the modified wood, so it was expected that samples with higher WPG should result in a higher durability (Krause and Militz 2004). This expectation was confirmed for the resistance against degradation caused by decay fungi and subterranean termites for the samples with 17% DMDHEU in the range of low WPG. The WPG of the D17T140 sample was too low to provide a lasting effect. From the present data, the influence of the different DMDHEU concentrations on biological durability cannot be determined. However, variations with similar DMDHEU concentrations but different curing temperatures led to significant differences.

The increase of curing temperature from 140 to 160 °C led to a significantly improved resistance, whereas further extending the temperature to 180 °C only had a poor effect. The results suggest that the degree of curing is different for the different curing temperatures employed. The curing is based on a condensation reaction of the modifying agent and hydroxyl groups of the cell wall components. A low curing temperature will hinder the condensation reaction and lead to a low degree of curing. Insufficient curing will allow leaching of the agents under the application conditions. This effect prohibits lasting protection of the OPT modified by the curing temperature of 140 °C. In contrast, the curing condition of 160 °C allows a high degree of curing, leading to effective protection against biological decay. While cross-linking improves stability, excessive heat from curing temperature of 180 °C degraded both the DMDHEU and the wood structure, leading to increased vulnerability to fungal and termite attacks. With respect to resistance against termite attack variation, D34T160 performed best, whereas the protection was insufficient for the variants D17T160. For some samples of variations, an initial slight attack through termites was observed in D34T160. However, during the duration of the test, the samples did not deteriorate further. It can be concluded that using the appropriate amount of DMDHEU and suitable curing temperature on the OPT modification leads to an improvement in the resistance against termites.

## CONCLUSIONS

1. Modification of the oil palm trunk (OPT) with the water-soluble glyoxal resin, 1,3-dimethylol-4,5-dihydroxy-ethyleneurea (DMDHEU) had a significant impact on the growth of molds, decay fungi, and subterranean termites, leading to improved resistance against biological degradation under laboratory conditions.
2. Increasing the curing temperature from 140 to 160 °C optimized the chemical reactions of DMDHEU, leading to stronger bonding within the OPT and improved durability. However, curing oil palm trunk at excessively high temperatures of 180 °C led to a decrease in biological durability which could be due to the degradation of DMDHEU and wood components, outweighing the benefits of cross-linking.
3. Ensuring a uniform dispersion of DMDHEU on modified OPT is crucial for effectively preventing degradation by wood-destroying fungi and termites in non-biocidal wood modification technologies such as DMDHEU.
4. The DMDHEU-modified OPT showed enhanced resistance against molds and decay fungi; however, its effect on subterranean termite attacks was comparatively less pronounced. Oil palm trunk modified with 34% DMDHEU and cured at 160 °C exhibited superior efficacy in minimizing mass loss attributed to biological degradation when compared to untreated controls.



5. These findings provide valuable insights for technologists aiming to simplify experimental designs concerning combined modifications using both DMEDEHU application and thermal treatment at elevated curing temperatures.
6. Nevertheless, further investigation into the quantity of cross-linkages achieved through different modification regimens is warranted. It is recommended to further the research on leaching of DMDHEU-modified OPT, to understand the prevailing effect of DMDHEU on the biological durability of OPT, whether it is due to cross-linking or filler dispersion.

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